National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2234

Gallium for Thermal Analysis

This Standard Reference Material (SRM) is intended for use in calibrating differential scanning calorimeters (DSC), differential thermal analyzers, and similar instruments. A unit of SRM 2234 consists of a small ingot (approximately 2 g) of high purity gallium (99.999 99 %), sealed in a polyethylene vial. The enthalpy of fusion and the fusion temperature were measured in an adiabatic calorimeter. Complete details of the measurements are given in reference 1.

Enthalpy of Fusion ($J \cdot g^{-1}$)	Fusion Temperature (K)
80.097 ± 0.032	302.9146 ± 0.0001

The certified values were obtained from a set of 35 adiabatic calorimetric measurements in the temperature range of 288.6 K to 314.35 K. The methods used for determination of the certified values are given in reference 1. The fusion temperature is the freezing point temperature on the International Temperature Scale of 1990.

The average of three determinations of the enthalpy of fusion, after adjusting the measured enthalpy increments for the pre-fusion and post-fusion enthalpy increment contributions, was 80.097 $J \cdot g^{-1}$. The standard deviation calculated from the three measurements was 0.0057 $J \cdot g^{-1}$, and the standard deviation of the mean was 0.0033 $J \cdot g^{-1}$. A coverage factor of 4 was used to calculate the uncertainty of the value of the enthalpy change due to fusion, ± 0.013 $J \cdot g^{-1}$, which corresponded to ± 0.016 % of the enthalpy of fusion. To this quantity, estimated uncertainties of the extrapolations of the crystal-phase enthalpy function, $\Delta H_{cr}(T_1 \rightarrow 302.9146\ \text{K})$, and of the calculated liquid-phase enthalpy increments, $\Delta H_l(302.9146\ \text{K} \rightarrow T_2)$, were added. To estimate these uncertainties, twice the root-mean-square (rms) deviation for the representations of the enthalpy increment measurements of the liquid and crystal phases, ± 0.07 %, was used to estimate the uncertainty of the sum of the pre-fusion and post-fusion enthalpy increments. This quantity is approximately ± 0.019 $J \cdot g^{-1}$ and corresponds to ± 0.024 % of the enthalpy of fusion. The combination of these uncertainties gives ± 0.032 $J \cdot g^{-1}$, which corresponds to a 95 % confidence interval [3].

Expiration of Certification: The certification of this SRM is valid until **01 August 2010**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur which affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification were performed by D.G. Archer of the NIST Physical and Chemical Properties Division.

Gregory Rosasco, Chief
Physical and Chemical Properties Division

Gaithersburg, MD 20899
Certificate Issue Date: 17 September 2004

Robert Watters, Jr., Acting Chief
Measurement Services Division

The technical and support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

Sample-enclosure Devices: Commercially available sample-enclosure devices for differential scanning calorimetry or differential thermal analysis are fabricated from a variety of sources such as aluminum, gold, alumina, and steel. Of these commercial sample-holder materials, aluminum is used most commonly because of low cost and good thermal characteristics. Gallium alloys rapidly with aluminum and its alloys. However, some aluminum sample holders that have been subjected to a heat treatment (placed in an oven at 843 K for more than one hour) show resistance to alloying that allows use of the gallium for a short period of time. Note that this heat treatment procedure works with some, not all, sample holders from manufacturers. Some aluminum sample-holder components have acquired a brownish color after or while being subjected to the heat treatment; these components invariably reacted with an enclosed sample of gallium. Anodization of aluminum pans is not the same thing as the heat treatment described above.

Use: Specific instructions for use, other than those specified above regarding the stabilization of aluminum DSC pans prior to use with this material, depend on the calibration protocol being used. Reference 2 is one such protocol.

Storage Instructions: When not in use, store SRM 2234 in the packaging provided or in a manner that provides equivalent or better protection against loss or damage.

REFERENCES

[1] Archer, D.G.; *The Enthalpy of Fusion of Gallium*; J. Chem. Eng. Data, Vol. 47, pp 304-309 (2002).
[2] ASTM E967-03; *Standard Practice for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers*; Annu. Book ASTM Stand., Vol. 14.02 (2003).
[3] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.